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(12) **PATENT APPLICATION (KOKKAI) (A) Hei-Sei 6-11607**

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Verification: not requested

Number of pages (11)

(54) Name of the invention: Manufacture of polymer interference films (Manufacture of diffraction lattices)

(21) Filed Number: Hei-Sei 4-170836

(22) Filed date: Hei-Sei 1 (1992) 6/29

Patent Assignee: Dainippon Printing Co.

JP 6-11607

[Note : Names, addresses, Company names and brand names are translated in the most common manner. Japanese Language does not have singular or plural words unless otherwise specified with numeral prefix or general form of plurality suffix. Translator's note.]

(54) [Name of the invention]

Manufacture of polymer interference films (manufacture of diffraction lattice)

(57) [Summary]

[Goal]

To manufacture a diffraction lattice where a diffraction is possible over a wide range of wavelengths, by the fact that two types and more polymer layers, that have different refraction indices, are laminated as in one part the thickness of the space between them is varied.

[Structure]

It is manufactured as a layer that is formed from 2 or more polymers with different refraction indices so that the refraction indices are repeated in the order :relatively high - low. And the laminated film is obtained as the high refraction index polymer layers 1, 3, 5 and the low refractive index polymer layers 2,4 6, are coated in that order. Because of the fact that it is a laminated layer film where the thickness of the space between the layers, is varied, it is possible to have a range of the refracted wavelengths, for example, in the range of 800 nm ~ 2000 nm. In this case, it becomes a good heat rays reflecting layer, which is permeable to the visible light, but reflects the heat rays. Besides that, there are also other applications like color filters where a light with specific wavelength from the visible light region, is reflected, film cutting off the ultra-violet rays etc..

[Range of the claims of the invention]

[Claim 1]

Diffraction lattice manufacturing method, characterized by the fact that, it is manufactured as a layer that is formed from 2 or more polymers with different refraction indices so that the refraction indices are repeated in the

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order :relatively high - low. And the laminated film is obtained as the high refraction index polymer layers and the low refractive index polymer layers are coated in that order and laminated.

[Claim 2]

Manufacturing method for the preparation of a diffraction lattice, according to the above described Claim 1, characterized by the fact that it is manufactured as the polymer layer thickness is different from at least one part of the space between the layers, and it is possible to have a diffraction over a wide range of wavelengths.

[Claim 3]

Manufacturing method for the preparation of a diffraction lattice, according to the above described Claim 1, and Claim 2, characterized by the fact that the polymer layers are coated by using a dissolved polymer solution, where it is dissolved in a solvent that can be eliminated by heating and that does not dissolve the polymer layers.

[Claim 4]

Manufacturing method for the preparation of a diffraction lattice, according to the above described Claim 1, and Claim 2, characterized by the fact that the polymer layers are coated by coating of an ionized radiation beam curable solution, capable of forming a polymer layer by irradiation with an ionized radiation beam.

[Claim 5]

Manufacturing method for the preparation of a diffraction lattice according to the above described Claims from 1 to 4, characterized by the fact that as the coating method spin-coating is used.

[Detailed description of the invention]

[0001]

[Technological sphere of application]

The present invention is an invention about the manufacturing method of a diffraction lattice. And especially, it is an invention about the manufacturing method of a diffraction lattice, like a thermal radiation reflecting layer, that diffracts over a wide range of wavelengths.

[0002]

[Previous technology]

As diffraction lattices, according to the previous technology, the Bragg diffraction lattices are well known where in layers of photo-polymers, bichromate - gelatine, silver salts etc., the light interference image is recorded and formed.

However, regarding these lattices, in all cases, the range of the diffraction wavelengths, is narrow, and constructions that are capable of diffracting over a wide range of wavelengths, have not been obtained.

[0003]

In order to use such Bragg diffraction lattice as a thermal rays reflecting layer etc., it is necessary that the diffraction wavelength is accomplished within a range of over several hundred nm.

[0004]

[Problems solved by the present invention]

The present invention, is an invention that is based on such considerations of the circumstances, and its goal is to suggest a manufacturing method for the preparation of a Bragg diffraction lattice where a diffraction is possible over a wide range of wavelengths. This is accomplished by the fact that two types and more polymer layers, are laminated as in one part the thickness of the space between the layers is varied.

[0005]

[Measures in order to solve the problems]

According to the manufacturing method of the present invention, in order to solve the above described problems, it is a manufacturing method for the preparation of a polymer layer where polymer layers with different refractive indices are laminated and where at least in one part the thickness of the space between the layers is different. And in this method, as the coating methods, spin-coating method is used and the layer thickness control is accomplished by the change in the viscosity of the coating solution and the number of revolutions per minute (rpm) during the spin coating process. Because of that, the diffraction coefficient and the diffraction wavelength, can be set freely by varying the polymer layer thickness, the number of the laminated layers, the difference between the refraction coefficients. And it is a very simple, for the manufacturing of diffraction lattices with a wide range of applications.

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[0002]

[0006]

Usually, if the incident angle is denoted as Θ , and the refraction coefficient - as n , the lattice space pitch, d , and the diffraction wavelength λ , are according to the following

$$2nd \sin \Theta = m \quad (m = 1, 2, 3, \dots)$$

Consequently, if the structure of the diffraction lattice is formed as a layer with a high refraction coefficient and a layer with a low refraction coefficient are laminated opposite to each other, at this time, by manufacturing several types of layers that have different lattice space pitch d , it is possible to realize a diffraction of different wavelength light. And it is possible to widen the diffraction wavelength range of one diffraction lattice.

[0007]

If we are to explain an example in more detail, in the case when the average refractive coefficient is 1.5, as it is shown in the sectional view diagram in Figure 1, for example, 5 combinations of a 0.13 micron thick high refraction coefficient polymer layer 1 ($nd = 1.59$) and the same thickness, low refraction coefficient polymer layer 2 ($nd = 1.41$) are laminated; and after that 5 combinations of 0.14 micron thick high refraction coefficient polymer layer 3 ($nd = 1.59$) and the same thickness low refraction coefficient polymer layer 4 ($nd = 1.41$) are laminated; and the layer lamination is repeated as the thickness is progressively increased, and 5 combinations of 0.67 micron thick high refraction coefficient polymer layer 5 ($nd = 1.59$) and the same thickness low refraction coefficient polymer layer 6 ($nd = 1.41$) are laminated and the diffraction lattice 10, is manufactured. The film thickness of this film 10, became 70 microns, as calculated according to the following calculations:

[0008]

$$0.13 \times 2 \times 5 + 0.14 \times 2 \times 5 + \dots + 0.67 \times 2 \times 5 = 70 \text{ microns}$$

By using this diffraction lattice 10, as it is shown in Figure 2, by the first thickness layer combination, 1 and 2, light with a wavelength of 800 nm, is diffracted, and by the combinations of the progressively increasing in thickness layers, a diffraction is accomplished over a wide range of 840 nm,, 2000 nm. Then, by varying the polymer layer thickness, the number of the laminated layers, and the difference in the refraction coefficient, it is possible to set freely the diffraction wavelength range and the diffraction coefficient.

[0009]

As methods in order to obtain such a layered structure, different coating processes can be considered, like, roll coating, gravure coating, die coating, spray coating, blade coating, spin coating etc., and each of this coating processes can be appropriately used in the present invention. Among those, as an especially preferred coating method, there is the spin coating method where the layer thickness control is 0.01 micron units, and also it is a very strict control.

[0010]

Also, regarding the polymers that form the polymer layers according to the present invention, it is preferred that they are coated by the spin coating method by using polymer solutions or ionization radiation beam curable type material solution.

[0011]

First, here below the case when it is coated by using a polymer solution, will be explained. In the case when it is coated by using a polymer solution, it is necessary that each of the described here below conditions is satisfied.

- (1) Regarding the polymer, it is dissolved in a solvent that after the spin coating can be heated and dried off.
- (2) The refraction coefficients of the two polymers are mutually different.
- (3) the solvent that dissolves one of the polymers, does not dissolve the layer of the other polymer.

As polymer compositions that satisfy the above described conditions, those can be determined as the polymer handbook etc., is used, and the refraction coefficients, solvents and nonsolvents are studied.

[0012]

As the polymers that can be used in the manufacturing method according to the present invention, for example,

polyvinylidene fluoride	1.42	
polydimethylsilylene (polydimethylsiloxane)		1.43
polytrifluoroethylmethacrylate	1.437	
polyoxypropylene	1.4495	
polyvinylisobutylether	1.4507	
polyvinylethylether		1.4540
polyoxyethylene	1.4563	

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[0009]

polyvinylbutylether	1.4563
polyvinylpentylether	1.4581
polyvinylhexylether	1.4591
poly(4-methy-1-pentene)	1.459 ~ 1.465
cellulose acetate butylate	1.46 ~ 1.49
poly(4-fluoro-2-trifluoromethylstyrene)	1.46
polyvinylloctylether	1.4613
poly(vinyl 2-ethylhexylether)	1.4626
polyvinyldecylether	1.4628
poly(2-methoxyethylacrylate)	1.463
polybutylacrylate	1.4631
polybutylacrylate	1.466
poly(t-butylmethacrylate)	1.4638
polyvinyl dodecylether	1.4640
poly (3-ethoxypropylacrylate)	1.465
polyoxycarbonyltetramethylene	1.465
polyvinylpropionate	1.4665
polyvinylacetate	1.4665
polyvinylmethylether	1.467
polyethylacrylate	1.4685
ethylene-vinyl acetate copolymer (80 % ~ 20 % vinylacetate)	1.47 ~ 1.50
cellulose propionate	1.47 ~ 1.49
cellulose acetate propionate	1.47
benzylcellulose	1.47 ~ 1.58
phenol formaldehyde resin	1.47 ~ 1.70
cellulose triacetate	1.47 ~ 1.48
polyvinylmethylether (isotactic)	1.4700
poly (3-methoxypropylacrylate)	1.471
poly(2-ethoxyethylacrylate)	1.471
polymethylacrylate	1.472 ~ 1.480
polyisopropylemethacrylate	1.4728
poly (1-decene)	1.4730
polypropylene (atactic, density 0.8575 g/cm ³)	1.4735
poly(vinyl sec-butylether) (isotactic)	1.4740
polydodecylmethacrylate	1.4740
polyoxyethyleneoxysuccinoyl (polyethylenesuccinate)	1.4744
polytetradecylmethacrylate	1.4746
ethylene - propylene copolymer material (EPR rubber)	1.4748 ~ 1.48
polyhexadecylmethacrylate	1.4750
polyvinylformate	1.4757
poly(2-fluoroethylmethacrylate)	1.4768
polyisobutylmethacrylate	1.477
ethyl cellulose	1.479
polyvinylacetal	1.48 ~ 1.50

cellulose acetate	1.48 ~ 1.50	
cellulose tripropionate	1.48 ~ 1.49	
polyoxymethylene	1.48	
polyvinylbutyral	1.48 ~ 1.49	
poly (n-hexylmethacrylate)		1.4813
poly (n-butylmethacrylate)		1.483
polyethylidenedimethacrylate	1.4831	
poly (2-ethoxyethylmethacrylate)		1.4833
polyoxyethyleneoxymaleoyl (polyethylene maleate)		1.4840
poly (n-propylmethacrylate)		1.484
poly (3, 3, 5-trimethylcyclohexylmethacrylate)		1.485
polyethylmethacrylate	1.485	
poly(2-nitro-2-methylpropylmethacrylate)		1.4868
polytriethylcarbinylmethacrylate		1.4889
poly (1,1'-diethylpropylmethacrylate)	1.4889	
polymethylmethacrylate	1.4893	
	1.490	
poly (2-decyl-1,3-butadiene)		1.4899
polyvinyl alcohol	1.49 ~ 1.53	
polyethylglycolate methacrylate	1.4903	
poly (3-methylcyclohexylmethacrylate)		1.4947
poly(cyclohexyl alpha-ethoxyacrylate)		1.4969
methylcellulose (low density)	1.497	
poly(4-methylcyclohexylmethacrylate)	1.4975	
polydecamethyleneglycoldimethacrylate		1.4990
polyurethane		1.5 ~ 1.6
poly (1,2-butadiene)		1.5000
polyvinylformal	1.50	
poly (2-bromo-4-trifluoromethylstyrene)		1.5
cellulose nitrate	1.50 ~ 1.514	
poly (sec-butyl alpha-chloroacrylate)	1.500	
poly (2-heptyl-1,3-butadiene)	1.5000	
poly (ethyl-alpha-chloroacrylate)		1.502
poly (2-isopropyl- 1,3-butadiene)		1.5028
poly (2-methylcyclohexylmethacrylate)		1.5028
polypropylene(density 0.9075 g/cm3)	1.5030	
polyisobutene	1.505 ~ 1.51	
polyborunylmethacrylate		1.5059
poly (2-t-butyl-1,3-butadiene)	1.5060	
polyethyleneglycoldimethacrylate		1.5063
polycyclohexylmethacrylate		1.5066
poly (cyclohexanediol -1,4-dimethacrylate)		1.5067
butyl rubber (not vulcanized)	1.508	
polytetrahydrofurfurylmethacrylate)	1.5096	
gudaperca (beta)	1.509	

cellulose acetate	1.48 ~ 1.50	
cellulose tripropionate	1.48 ~ 1.49	
polyoxymethylene	1.48	
polyvinylbutyral	1.48 ~ 1.49	
poly (n-hexylmethacrylate)		1.4813
poly (n-butylmethacrylate)		1.483
polyethylidenedimethacrylate	1.4831	
poly (2-ethoxyethylmethacrylate)		1.4833
polyoxyethyleneoxymaleoyl (polyethylene maleate)		1.4840
poly (n-propylmethacrylate)		1.484
poly (3, 3, 5-trimethylcyclohexylmethacrylate)		1.485
polyethylmethacrylate	1.485	
poly(2-nitro-2-methylpropylmethacrylate)		1.4868
polytriethylcarbinylmethacrylate		1.4889
poly (1,1'-diethylpropylmethacrylate)	1.4889	
polymethylmethacrylate	1.4893	
	1.490	
poly (2-decyl-1,3-butadiene)		1.4899
polyvinyl alcohol	1.49 ~ 1.53	
polyethylglycolate methacrylate	1.4903	
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poly(4-methylcyclohexylmethacrylate)	1.4975	
polydecamethyleneglycoldimethacrylate		1.4990
polyurethane		1.5 ~ 1.6
poly (1,2-butadiene)		1.5000
polyvinylformal	1.50	
poly (2-bromo-4-trifluoromethylstyrene)		1.5
cellulose nitrate	1.50 ~ 1.514	
poly (sec-butyl alpha-chloroacrylate)	1.500	
poly (2-heptyl-1,3-butadiene)	1.5000	
poly (ethyl-alpha-chloroacrylate)		1.502
poly (2-isopropyl- 1,3-butadiene)		1.5028
poly (2-methylcyclohexylmethacrylate)		1.5028
polypropylene(density 0.9075 g/cm ³)	1.5030	
polyisobutene	1.505 ~ 1.51	
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polyvinylmethylether	1.467
polyethylacrylate	1.4685
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poly(1-decene)	1.4730
polypropylene (atactic, density 0.8575 g/cm ³)	1.4735
poly(vinyl sec-butylether) (isotactic)	1.4740
polydodecylmethacrylate	1.4740
polyoxyethyleneoxysuccinoyl (polyethylenesuccinate)	1.4744
polytetradecylmethacrylate	1.4746
ethylene - propylene copolymer material (EPR rubber)	1.4748 ~ 1.48
polyhexadecylmethacrylate	1.4750
polyvinylformate	1.4757
poly(2-fluoroethylmethacrylate)	1.4768
polyisobutylmethacrylate	1.477
ethyl cellulose	1.479
polyvinylacetal	1.48 ~ 1.50

polyethylene ionomer	1.51
polyoxyethylene (high molecular weight)	1.51 ~ 1.54
polyethylene (density 0.914 g/cm ³)	1.51
(density 0.94 ~ 0.945 g/cm ³)	1.52 ~ 1.53
(density 0.965 g/cm ³)	1.545
poly (1-methylcyclohexylmethacrylate)	1.5111
poly (2-hydroxyethylmethacrylate)	1.5119
polyvinylchloroacetate	1.512
polybutene (isotactic)	1.5125
polyvinylmethacrylate	1.5129
poly (N-butyl-methacrylamide)	1.5135
gutaperca (alpha)	1.514
terpenic resin	1.515
poly (1,3-butadiene)	1.5154
shelac	1.51 ~ 1.53
poly (methyl alpha-chloroacrylate)	1.517
poly (2-chloroethylmethacrylate)	1.517
poly (2-diethylaminoethylmethacrylate)	1.5174
poly (2-chlorocyclohexylmethacrylate)	1.5179
poly (1,3-butadiene) (35 % cis; 56 % trans; 7 %, 2- contet)	1.5180
natural rubber	1.519 ~ 1.52
polyallylmethacrylate	1.5196
polyvinyl chloride + 40 % octylphthalate	1.52
polyacrylonitrile	1.52
	1.5187
polymethacrylonitrile	1.52
poly (1, 3-butadiene) (sic type rich)	1.52
butadiene - acrylonitrile copolymer material	1.52
polymethylisopropenyl ketone	1.5200
polyisoprene	1.521
polyester resin rigid (approximately 50 % styrene)	1.523 ~ 1.54
poly (N- (2-methoxyethyl)methacrylamide)	1.5246
poly (2,3-dimethylbutadiene) (methyl rubber)	1.525
vinyl chloride - vinyl acetate copolymer material (95/5 ~ 90/10)	1.525 ~ 1.535
polyacrylic azide	1.527
poly (1,3-dichloropropylmethacrylate)	1.5270
poly (2-chloro-1- (chloromethyl) ethylmethacrylate)	1.5270
polyacroleine	1.529
poly (1-vinyl -2- pyrrolidone)	1.53
hydrochloric acid reacted rubber	1.53 ~ 1.55
Nylon 6; Nylon 6,6; Nylon 6, 10 (synthetic type material)	1.53
(Nylon 6-fiber; 1.515 in the transverse direction; 1.565 in the fiber direction)	
butadiene - styrene copolymer material (approximately 30 % styrene)	1.53
block copolymer	
poly (cyclohexyl alpha-chloroacrylate)	1.532

poly (2-chloroethyl alpha-chloroacrylate)	1.533
butadiene-styrene copolymer material (approximately 75/25)	1.535
poly (2-aminoethylmethacrylate)	1.537
polyfurfurylmethacrylate	1.5381
protein	1.539 ~ 1.541
polybutylmercaptylmethacrylate	1.5390
poly (1-phenyl-n-amylmethacrylate)	1.5396
poly (N-methyl-methacrylamide)	1.5398
cellulose	1.54
polyvinylchloride	1.54 ~ 1.55
urea- formaldehyde resin	1.54 ~ 1.56
poly (sec-butyl alpha-bromoacrylate)	1.542
poly (cyclohexyl alpha-bromoacrylate)	1.542
poly (2-bromoethylmethacrylate)	1.5426
polydihydroabietic acid	1.544
polyabietic acid	1.546
polyethylmercaptylmethacrylate	1.547
poly (N-allylmethacrylamide)	1.5476
poly (1-phenylethylmethacrylate)	1.5487
polyvinylfuran	1.55
poly (2-vinyltetrahydrofuran)	1.55
poly (vinyl chloride) + 40 % tricrezyl phosphate	1.55
epoxy resin	1.55 ~ 1.60
poly (p-methoxybenzylmethacrylate)	1.552
polyisopropylmethacrylate	1.552
poly (p-isopropylstyrene)	1.554
polychloroprene	1.554 ~ 1.558
poly (oxyethylene-alpha-benzoate-methacrylate)	1.555
poly (p,p'-xylilenyldimethacrylate)	1.5559
poly (1-phenylallylmethacrylate)	1.5573
poly(p-cyclohexylphenylmethacrylate)	1.5575
poly (2-phenylethylmethacrylate)	1.5592
poly (oxycarbonyloxy -1,4-phenylene-1-propyl-butylidene 1,4-phenylene)	1.5602
poly (1-(o-chlorophenyl)ethylmethacrylate)	1.5624
styrene - anhydrous maleic acid copolymer material	1.564
poly (1-phenylcyclohexylmethacrylate)	1.5645
poly (oxycarbonyloxy-1,4-phenylene 1,3-dimethyl-butylidene - 1,4-phenylene)	1.5671
poly (methyl alpha - bromoacrylate)	1.5672
polybenzylmethacrylate	1.5680
poly (2-(phenylsulfonyl)ethylmethacrylate)	1.5682
poly(m-crezylmethacrylate)	1.5683
styrene - acrylonitrile copolymer material (approximately 75/25)	1.57
poly (oxycarbonyloxy-1,4-phenyleneisobutylidene -1,4-phenylene)	1.5702
poly (o-methoxyphenylmethacrylate)	1.5705

poly (2-chloroethyl alpha-chloroacrylate)	1.533
butadiene-styrene copolymer material (approximately 75/25)	1.535
poly (2-aminoethylmethacrylate)	1.537
polyfurfurylmethacrylate	1.5381
protein	1.539 ~ 1.541
polybutylmercaptylmethacrylate	1.5390
poly (1-phenyl-n-amylnmethacrylate)	1.5396
poly (N-methyl-methacrylamide)	1.5398
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polyvinylchloride	1.54 ~ 1.55
urea- formaldehyde resin	1.54 ~ 1.56
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poly (1-phenylethylmethacrylate)	1.5487
polyvinylfuran	1.55
poly (2-vinyltetrahydrofuran)	1.55
poly (vinyl chloride) + 40 % tricrezyl phosphate	1.55
epoxy resin	1.55 ~ 1.60
poly (p-methoxybenzylmethacrylate)	1.552
polyisopropylmethacrylate	1.552
poly (p-isopropylstyrene)	1.554
polychloroprene	1.554 ~ 1.558
poly (oxyethylene-alpha-benzoate- -methacrylate)	1.555
poly (p,p'-xylilenyldimethacrylate)	1.5559
poly (1-phenylallylmethacrylate)	1.5573
poly(p-cyclohexylphenylmethacrylate)	1.5575
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poly (o-methoxyphenylmethacrylate)	1.5705

polyethylene ionomer	1.51
polyoxyethylene (high molecular weight)	1.51 ~ 1.54
polyethylene (density 0.914 g/cm ³)	1.51
(density 0.94 ~ 0.945 g/cm ³)	1.52 ~ 1.53
(density 0.965 g/cm ³)	1.545
poly (1-methylcyclohexylmethacrylate)	1.5111
poly (2-hydroxyethylmethacrylate)	1.5119
polyvinylchloroacetate	1.512
polybutene (isotactic)	1.5125
polyvinylmethacrylate	1.5129
poly (N-butyl-methacrylamide)	1.5135
gutaperca (alpha)	1.514
terpenic resin	1.515
poly (1,3-butadiene)	1.5154
shelac	1.51 ~ 1.53
poly (methyl alpha-chloroacrylate)	1.517
poly (2-chloroethylmethacrylate)	1.517
poly (2-diethylaminoethylmethacrylate)	1.5174
poly (2-chlorocyclohexylmethacrylate)	1.5179
poly (1,3-butadiene) (35 % cis; 56 % trans; 7 %, 2- contet)	1.5180
natural rubber	1.519 ~ 1.52
polyallylmethacrylate	1.5196
polyvinyl chloride + 40 % octylphthalate	1.52
polyacrylonitrile	1.52
	1.5187
polymethacrylonitrile	1.52
poly (1, 3-butadiene) (sic type rich)	1.52
butadiene - acrylonitrile copolymer material	1.52
polymethylisopropenyl ketone	1.5200
polyisoprene	1.521
polyester resin rigid (approximately 50 % styrene)	1.523 ~ 1.54
poly (N- (2-methoxyethyl)methacrylamide)	1.5246
poly (2,3-dimethylbutadiene) (methyl rubber)	1.525
vinyl chloride - vinyl acetate copolymer material (95/5 ~ 90/10)	1.525 ~ 1.535
polyacrylic azide	1.527
poly (1,3-dichloropropylmethacrylate)	1.5270
poly (2-chloro-1- (chloromethyl) ethylmethacrylate)	1.5270
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butadiene - styrene copolymer material (approximately 30 % styrene)	1.53
block copolymer	
poly (cyclohexyl alpha-chloroacrylate)	1.532

polyphenylmethacrylate	1.5706
poly (o-crezylmethacrylate)	1.5707
polydiallylphtalate	1.572
poly (2,3-dibromopropylmethacrylate)	1.5739
poly (oxycarbonyloxy - 1,4-phenylene -1-methyl-butyldiene-1,4-phenylene)	1.5745
poly (oxy-2,6-dimethylphenylene)	1.575
polyoxyethyleneoxyterephthaloyl (amorphous)	1.5750
(polyethylene terephthalate)	
(crystalline fiber: 1.51 in the transverse direction 1.64 in the direction of the fiber)	
polyvinyl benzoate	1.5775
poly(oxycarbonyloxy -1,4-phenylene butyldiene-1,4-phenylene)	1.5792
poly (1,2-diphenyl ethylmethacrylate)	1.5816
poly (o-chlorobenzylmethacrylate)	1.5823
poly(oxycarbonyloxy - 1,4-phenylene -sec-butyldiene-1,4-phenylene)	1.5827
polyoxypentaerithritoxyphtaloyl)	1.584
poly (m-nitrobenzylmethacrylate)	1.5845
poly(oxycarbonyloxy -1,4- phenyleneisopropylidene-1,4-phenylene)	1.5850
poly (N-(2-phenylethyl) methacrylamide)	1.5857
poly(4-methoxy--2- methylstyrene)	1.5868
poly (o-methylstyrene)	1.5874
polystyrene	1.59 ~ 1.592
poly(oxycarbonyloxy-1,4-phenylenecyclohexylidene-1,4-phenylene)	1.5900
poly (o-methoxystyrene)	1.5932
polydiphenylmethylethylmethacrylate	1.5933
poly(oxycarbonyloxy-1,4-phenyleneethylidene-1,4-phenylene)	1.5937
poly (p-bromophenylmethacrylate)	1.5964
poly (N-benzylmethacrylamide)	1.5965
poly (p-methoxystyrene)	1.5967
hardened rubber (32 % S)	1.6
polyvinylidene chloride	1.60 ~ 1.63
polysulfide ("Thiokol")	1.6 ~ 1.7
poly (o-chlorodiphenylmethylethylmethacrylate)	1.6040
poly (oxycarbonyloxy-1,4- (2,6-dichloro) phenylene-isopropylidene - 1,4-(2,6-dichloro) phenylene)	1.6056
poly(oxycarbonyloxybis (1,4-(3,5-dichlorophenylene)))	1.6056
polypentachlorophenylmethacrylate	1.608
poly (o-chlorostyrene)	1.6098
poly(phenyl alpha- bromoacrylate)	1.612
poly(p-divinylbenzene)	1.6150

poly (N-vinylphtalimide)	1.6200
poly(2,6-dichlorostyrene)	1.6248
poly(beta-naphthylmethacrylate)	1.6298
poly(alpha-naphthylcarbinylnmethacrylate)	1.63
polysulfon	1.633
poly (2-vinylthiophen)	1.6376
poly (alpha-naphthylmethacrylate)	1.6410
poly (oxycarbonyloxy-1,4-phenylenediphenyl- methylene - 14,-phenylene)	1.6539
polyvinylphenylsulfide	1.6568
butylphenolformaldehyde resin	1.66
urea - thiourea- formaldehyde resin	1.660
polyvinylnaphthalene	1.6818
polyvinylcarbazol	1.683
naphthalene - formaldehyde resin	1.696
phenol -formaldehyde resin	1.70
polypentabromophenylmethacrylate	1.71
etc., can be used. Moreover, regarding the above described compounds, the d line refraction coefficient (nd) is also shown.	

[0013]

Also, as representative solvents, methanol, ethanol, n-propanol, isopropanol, amyl alcohol, acetone, methylethylketone, methylisobutylketone, ethylenechloride, chloroform, benzene, toluene, xylene, hexene, heptane, cyclohexane, etc., relatively low boiling point organic solvents and water can be used.

[0014]

The case when it is coated as the ionization radiation beam curable type material solution, is described here below. As the ionization radiation beam curable type material, there are the ionization radiation beam curable type resins and ultra-violet light curable type resins. And regarding these ionization radiation beam curable type resins and ultra-violet light curable type resins, except for the fact that in the latter, a polymerization initiation agent and a sensitivity increasing agent, are contained, the other components are the same. And usually, as a component of the cover layer forming material, there is a material whose main component is a polymer, oligomer, or monomer that contains in its structure a radical that is active in the radical polymerization reaction. And it is preferred that the viscosity of this material is less than 2000 cps. As such a polymer or oligomer material, materials that are easily available as commercial products, like urethane acrylate or polyester acrylate, can be appropriately used according to the present invention. As the monomer material, the commercially available acrylic acid or methacrylic acid derivative materials etc., ionization radiation beam curable type

poly (N-vinylphthalimide)	1.6200
poly(2,6-dichlorostyrene)	1.6248
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polypentachlorophenylmethacrylate	1.608
poly (o-chlorostyrene)	1.6098
poly(phenyl alpha- bromoacrylate)	1.612
poly(p-divinylbenzene)	1.6150

monomers, can be appropriately used according to the present invention. In order to make the above described curable resins ultra-violet light curable resins, in that, it is possible to use and mix as a light polymerization agent, acetophenone type, benzophenone, mihira- benzoylbenzoate, alpha-amineoximeester, tetramethylthiorame monosulfide, thioxantane etc., types, and as an agent increasing the light sensitivity,, it is possible to use and mix n-butylamine, triethylamine, tri-n-butylphosphine etc..

[0015]

In this case, by using spin coating, the above described material is coated at the desired thickness, and after that it is irradiated by using an ionized irradiation beam method. And it is possible to use the previous technology in the state as it is, for example, in the case of the electron beam radiation, the Kokuroft Walton type, Bandegraf type, resonance pressure change type, insulated core pressure change type, direct beam type, Dynamitron type, high frequency type, etc., it can be irradiated from different types of electron beam acceleration devices. And it is an electron beam that has an energy in the range of 50 ~ 1,000 KeV, and preferably, it is in the range of 100 ~ 300 KeV. And by using this beam in the range of 0.1 ~ 100 Mrad, and preferably, in the range of 1 ~ 10 Mrad, are irradiated and by that it is possible to cure the material. And also, in the case of the ultra-violet irradiation, by using the ultra-violet light beam that is generated from a light source like an ultra-high pressure mercury lamp, high pressure mercury lamp, carbon arc, xenone arc, metal hallide lamp etc., a radiation in the range of 0.1 ~ 10,000 mJ/cm², preferably, in the range of 10 ~ 1,000 mJ/cm², is generated and by that it is possible to accomplish the cure.

[0016]

Usually, in the spin coating method, the thickness, h, of the coating layer, is represented by

$$h = K \mu^{0.36} \omega^{-0.50} (E \eta / C_p)^{0.60}$$

Here, K : constant, μ : viscosity of the coating material, ω : angular velocity of the spinner, E : relative evaporation rate, η : evaporation hidden heat, C_p : heat capacity.

[0017]

Consequently, regarding the conditions for coating a polymer layer with a desired thickness, in advance, the polymer solution is adjusted to the desired viscosity and it is coated a the desired number of rpms, and by that the relationship between the layer thickness and the rpms, is established. And it is possible to determined the desired coating thickness from that relationship.

[0018]

As it is clear from the above described explanation, the manufacturing method for the preparation of the diffraction lattice according to the present invention, is a manufacturing method characterized by the fact that layers formed from 2 and more polymer layers with different refraction coefficients are repeated so that the relatively high and the relatively low refraction indices are repeated, and the high refraction index polymer layer and the low refraction index polymer layer are coated in order and by that they are laminated.

[0019]

In this case, the fact that the polymer layer thickness is made so that at least in one part is different from the layer space, is required so that a diffraction over a wide range of wavelengths can be conducted.

[0020]

Also, regarding the polymer layers, they are produced by coating a polymer solution where the polymer is dissolved in a solvent medium that can be eliminated by heating and drying and also that does not dissolve the polymer layers, or it is manufactured by coating an ionized radiation beam curable type solution, where the polymer layer can be formed by irradiation with an ionized radiation beam.

[0021]

In the case when the polymer solution is snapping etc., and by that it is difficult to accomplish a homogeneous coating, it is possible to solve this problem by using a surface treatment, like a corona treatment, ozone treatment etc..

[0022]

Moreover, it is preferred to use spin coating as the coating method.

[0023]

[Effect]

According to the present invention, it is a manufacturing method characterized by the fact that layers formed from 2 and more polymer layers with different refraction coefficients are repeated so that the relatively high and the relatively low refraction indices are repeated, and the high refraction

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According to the present invention, it is a manufacturing method characterized by the fact that layers formed from 2 and more polymer layers with different refraction coefficients are repeated so that the relatively high and the relatively low refraction indices are repeated, and the high refraction

monomers, can be appropriately used according to the present invention. In order to make the above described curable resins ultra-violet light curable resins, in that, it is possible to use and mix as a light polymerization agent, acetophenone type, benzophenone, mihira- benzoylbenzoate, alpha-amineoximeester, tetramethylthiorame monosulfide, thioxantane etc., types, and as an agent increasing the light sensitivity,, it is possible to use and mix n-butylamine, triethylamine, tri-n-butylphosphine etc..

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Consequently, regarding the conditions for coating a polymer layer with a desired thickness, in advance, the polymer solution is adjusted to the desired viscosity and it is coated a the desired number of rpms, and by that the relationship between the layer thickness and the rpms, is established. And it is possible to determined the desired coating thickness from that relationship.

index polymer layer and the low refraction index polymer layer are coated in order and by that they are laminated. And because of that, a laminated layer is obtained where the spaces between the layers are different and by that it is possible to easily manufacture a diffraction lattice with a wide range of diffraction wavelengths.

[0024]

[Practical example]

Here below, a practical example of the manufacturing method for the preparation of a diffraction lattice, according to the present invention, will be explained.

Practical Example 1

As a high refraction coefficient polymer layer, polystyrene (PSt, $n_d=1.592$) and as a low refraction coefficient polymer layer polyhydroxyethylmethacrylate (PHEMA, $n_d=1.46$) were used, and a multilayer polymer layer film was manufactured, where light with wavelengths in the range of 800 nm ~ 2000 nm, was diffracted, and it was formed as each layer thickness contained 20 combinations of high and low refractive coefficient polymer layers and by a 0.033 pitch layer thickness in the range of 0.129 microns ~ 0.327 microns space.

[0025]

Here, the polystyrene, which was a commercially available material with a molecular weight of 100,000 (manufactured by Denka Company), was prepared as a 5 % dioxane solution (viscosity : 10.0 cps), and the PHEMA was a synthesize material with a molecular weight of 100,000, and it was prepared as a 7 % methanol solution (viscosity : 12.3 cps), and they were coated. Moreover, for the PHEMA, it was synthesized by a radical polymerization as the concentration of the monomer in the methanol solution was 30 %, and 0.1 % of an initiation agent (V-65: manufactured by Shoko Junyaku Company) was used.

[0026]

For each of the coating thicknesses, the spin coating conditions of the PHEMA, are according to the shown here below table.

[0027]

膜厚 (μm)	高屈折率材 2 (r p m)	低屈折率材 3 (r p m)
0. 129	2500	2187
0. 162	1600	1400
0. 195	780	973
0. 228	816	714
0. 261	624	546
0. 294	492	430
0. 327	400	350

1. layer thickness

When the diffraction coefficient of the obtained diffraction lattice was measured by using an optical spectrophotometer (UV-365: manufactured by Shimadzu), it was understood that it maintained a homogeneous diffraction coefficient of 48 % over a range of 800 nm ~ 2000 nm.

[0028]

Practical Example 2

As the high refraction index polymer layer and as the low refraction index polymer layer, the shown here below ionized radiation curable type composition materials, were used.

[0029]

High refraction index polymer layer

urethane acrylate (PR-202:manufactured by Mitsubishi Kasei Company)

.....50 weight parts

2,4,5 -tribromophenolmethacrylate150 weight parts

(manufactured by Shoko Junyaku Industries)

dipentaerithritolpentaacrylate

(Satoma-399: manufactured by Satoma Company).....5 weight parts

viscosity : 10 cps, refraction coefficient: 1.58

Low refraction index polymer layer

urethane acrylate (PR-202:manufactured by Mitsubishi Kasei Company)

.....50 weight parts

(10)

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膜厚 (μm)	高屈折率材 2 (r p m)	低屈折率材 3 (r p m)
0. 129	2500	2187
0. 162	1800	1400
0. 195	780	973
0. 228	816	714
0. 261	624	546
0. 294	492	430
0. 327	400	350

1. layer thickness

When the diffraction coefficient of the obtained diffraction lattice was measured by using an optical spectrophotometer (UV-365: manufactured by Shimadzu), it was understood that it maintained a homogeneous diffraction coefficient of 48 % over a range of 800 nm ~ 2000 nm.

[0028]

Practical Example 2

As the high refraction index polymer layer and as the low refraction index polymer layer, the shown here below ionized radiation curable type composition materials, were used.

[0029]

High refraction index polymer layer

urethane acrylate (PR-202:manufactured by Mitsubishi Kasei Company)

.....50 weight parts

2,4,5 -tribromophenolmethacrylate

.....150

weight parts

(manufactured by Shoko Junyaku Industries)

dipentaerithritolpentaacrylate

(Satoma-399: manufactured by Satoma Company).....5 weight parts

viscosity : 10 cps, refraction coefficient: 1.58

Low refraction index polymer layer

urethane acrylate (PR-202:manufactured by Mitsubishi Kasei Company)

.....50 weight parts

index polymer layer and the low refraction index polymer layer are coated in order and by that they are laminated. And because of that, a laminated layer is obtained where the spaces between the layers are different and by that it is possible to easily manufacture a diffraction lattice with a wide range of diffraction wavelengths.

[0024]

[Practical example]

Here below, a practical example of the manufacturing method for the preparation of a diffraction lattice, according to the present invention, will be explained.

Practical Example 1

As a high refraction coefficient polymer layer, polystyrene (PSt, $n_d=1.592$) and as a low refraction coefficient polymer layer polyhydroxyethylmethacrylate (PHEMA, $n_d=1.46$) were used, and a multilayer polymer layer film was manufactured, where light with wavelengths in the range of 800 nm ~ 2000 nm, was diffracted, and it was formed as each layer thickness contained 20 combinations of high and low refractive coefficient polymer layers and by a 0.033 pitch layer thickness in the range of 0.129 microns ~ 0.327 microns space.

[0025]

Here, the polystyrene, which was a commercially available material with a molecular weight of 100,000 (manufactured by Denka Company), was prepared as a 5 % dioxane solution (viscosity : 10.0 cps), and the PHEMA was a synthesize material with a molecular weight of 100,000, and it was prepared as a 7 % methanol solution (viscosity : 12.3 cps), and they were coated. Moreover, for the PHEMA, it was synthesized by a radical polymerization as the concentration of the monomer in the methanol solution was 30 %, and 0.1 % of an initiation agent (V-65: manufactured by Shoko Junyaku Company) was used.

[0026]

For each of the coating thicknesses, the spin coating conditions of the PHEMA, are according to the shown here below table.

[0027]

2,2,3,3-tetrafluoropropylacrylate150 weight parts
 (commercial product, manufactured by Daikin Kasei Company)
 dipentaerithritolpentaacrylate
 (Satoma-399: manufactured by Satoma Company).....5 weight parts

viscosity : 8 cps, refraction coefficient: 1.47

The conditions of the spin coating for each of the different layer thicknesses of the high refractive index and the low refractive index materials, are shown according to the table here below.

[0030]

膜厚 (μm)	P S t (r p m)	P H E M A (r p m)
0. 1 2 9	6 2 5	1 3 3 1
0. 1 6 2	4 0 0	8 5 2
0. 1 9 5	2 7 8	5 9 2
0. 2 2 8	2 0 4	4 3 5
0. 2 6 1	1 5 6	3 3 2
0. 2 9 4	1 2 3	2 6 2
0. 3 2 7	1 0 0	2 1 3

1. layer thickness, 2. high refractive index material, 3. low refractive index material

Regarding each layer, after the spin coating, it was irradiated with 5 Mrad by using an electron beam (device manufactured by ESI company) and by that it was cured and a polymer layer was obtained. Regarding each of the layer thicknesses, the high refractive index layer and the low refractive index layer were mutually combined in 10 groups and laminated in order, and by that the diffraction lattice was manufactured.

[0031]

When the diffraction coefficient of the obtained diffraction lattice was measured by using an optical spectrophotometer (UV-365: manufactured by Shimadzu), it was understood that it maintained a homogeneous diffraction coefficient of 52 % over a range of 800 nm ~ 2000 nm.

[0032]

[Results from the invention]

As it is clear from the above described, according to the manufacturing method for the preparation of a diffraction lattice according to the present invention, it is manufactured as a layer that is formed from 2 or more polymers with different refraction indices so that the refraction indices are repeated in the order :relatively high - low. And the laminated film is obtained as the high refraction index polymer layers and the low refractive index polymer layers are coated in that order. Because of the fact that it is a laminated layer film where the thickness of the space between the layers, is varied, it is possible to have a range of the refracted wavelengths.

[0033]

In more detail, for example, it is possible to diffract at wavelengths in the range of 800 nm ~ 2000 nm. In this case, it becomes a good heat rays reflecting layer, which is permeable to the visible light, but reflects the heat rays. Such a layer reflecting the heat rays can be used in automobile and housing construction windows, and by that it is possible to reduce the increase of the temperature inside cars and houses.

[0034]

Also, besides that, there are also other applications like color filters, used in liquid crystal displays where a light with specific wavelength from the visible light region, is reflected, film cutting off the ultra-violet rays etc..

[Simple explanation of the figures]

[Figure 1]

It is a figure representing a sectional view of the laminated film of one detailed example of a diffraction lattice, manufactured according to the present invention.

[Figure 2]

It is a figure representing the diffraction properties of the diffraction lattice according to Figure 1.

[Explanation of the signs]

When the diffraction coefficient of the obtained diffraction lattice was measured by using an optical spectrophotometer (UV-365: manufactured by Shimazu), it was understood that it maintained a homogeneous diffraction coefficient of 52 % over a range of 800 nm ~ 2000 nm.

[0032]

[Results from the invention]

As it is clear from the above described, according to the manufacturing method for the preparation of a diffraction lattice according to the present invention, it is manufactured as a layer that is formed from 2 or more polymers with different refraction indices so that the refraction indices are repeated in the order :relatively high - low. And the laminated film is obtained as the high refraction index polymer layers and the low refractive index polymer layers are coated in that order. Because of the fact that it is a laminated layer film where the thickness of the space between the layers, is varied, it is possible to have a range of the refracted wavelengths.

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Also, besides that, there are also other applications like color filters, used in liquid crystal displays where a light with specific wavelength from the visible light region, is reflected, film cutting off the ultra-violet rays etc..

[Simple explanation of the figures]

[Figure 1]

It is a figure representing a sectional view of the laminated film of one detailed example of a diffraction lattice, manufactured according to the present invention.

[Figure 2]

It is a figure representing the diffraction properties of the diffraction lattice according to Figure 1.

[Explanation of the signs]

2,2,3,3-tetrafluoropropylacrylate150 weight parts
 (commercial product, manufactured by Daikin Kasei Company)
 dipentaerithritolpentaacrylate
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viscosity : 8 cps, refraction coefficient: 1.47

The conditions of the spin coating for each of the different layer thicknesses of the high refractive index and the low refractive index materials, are shown according to the table here below.

[0030]

膜厚 (μm)	P S t (r p m)	PHEMA (r p m)
0. 1 2 9	6 2 5	1 3 3 1
0. 1 6 2	4 0 0	8 5 2
0. 1 9 5	2 7 8	5 9 2
0. 2 2 8	2 0 4	4 3 5
0. 2 6 1	1 5 6	3 3 2
0. 2 9 4	1 2 3	2 6 2
0. 3 2 7	1 0 0	2 1 3

1. layer thickness, 2. high refractive index material, 3. low refractive index material

Regarding each layer, after the spin coating, it was irradiated with 5 Mrad by using an electron beam (device manufactured by ESI company) and by that it was cured and a polymer layer was obtained. Regarding each of the layer thicknesses, the high refractive index layer and the low refractive index layer were mutually combined in 10 groups and laminated in order, and by that the diffraction lattice was manufactured.

[0031]

- 1.....high refraction index polymer layer with a layer thickness of 0.13 microns ($n_d = 1.59$)
- 2.....low refraction index polymer layer with a layer thickness of 0.13 microns ($n_d = 1.41$)
- 3.....high refraction index polymer layer with a layer thickness of 0.14 microns ($n_d = 1.59$)
- 4.....low refraction index polymer layer with a layer thickness of 0.14 microns ($n_d = 1.41$)
- 5.....high refraction index polymer layer with a layer thickness of 0.67 microns ($n_d = 1.59$)
- 6.....low refraction index polymer layer with a layer thickness of 0.67 microns ($n_d = 1.41$)
- 10.....diffraction lattice (layer thickness 70 microns)

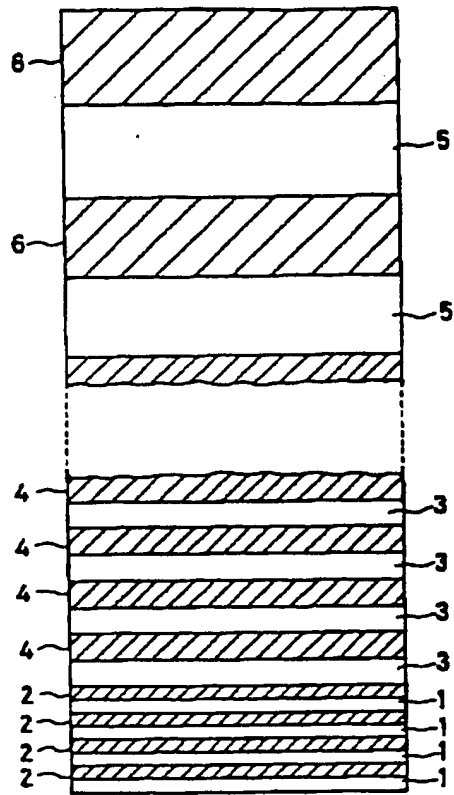
In Figure 2:

on the vertical axis - diffraction coefficient

on the horizontal axis - diffraction wavelength

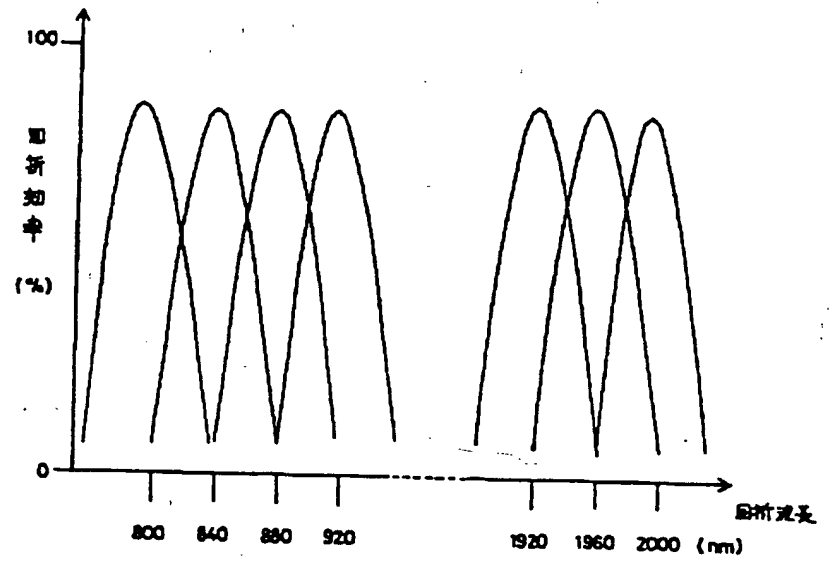
Patent Assignee: Dainippon Printing Company

【图1】

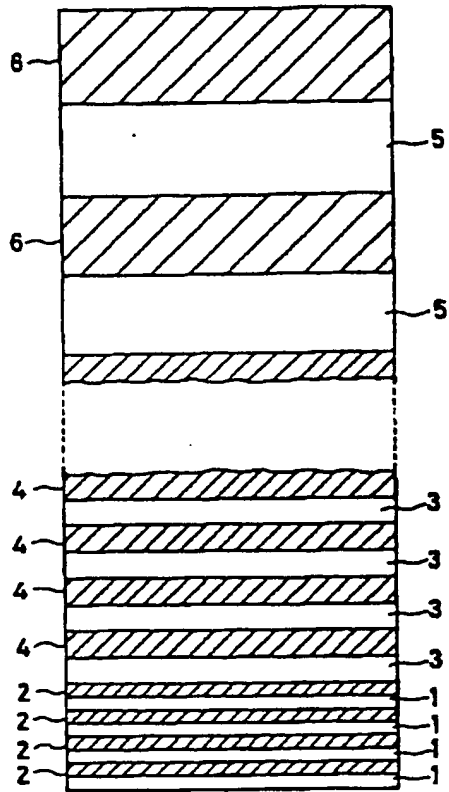


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【图2】

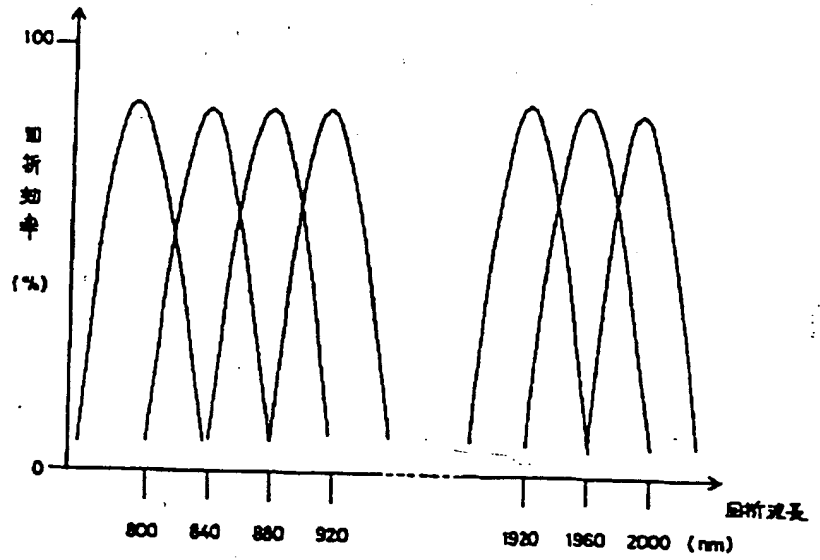


【图1】



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【图2】



- 1.....high refraction index polymer layer with a layer thickness of 0.13 microns ($n_d = 1.59$)
- 2.....low refraction index polymer layer with a layer thickness of 0.13 microns ($n_d = 1.41$)
- 3.....high refraction index polymer layer with a layer thickness of 0.14 microns ($n_d = 1.59$)
- 4.....low refraction index polymer layer with a layer thickness of 0.14 microns ($n_d = 1.41$)
- 5.....high refraction index polymer layer with a layer thickness of 0.67 microns ($n_d = 1.59$)
- 6.....low refraction index polymer layer with a layer thickness of 0.67 microns ($n_d = 1.41$)
- 10.....diffraction lattice (layer thickness 70 microns)

In Figure 2:

on the vertical axis - diffraction coefficient

on the horizontal axis - diffraction wavelength

Patent Assignee: Dainippon Printing Company

Translated by Albena Blagev (6-7946)

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